AUTOCLAVE CORROSION INHIBITOR EVALUATION

Cormetrics Job #: 12-123

Prepared for: ABC Company
1. Introduction

Four batch corrosion inhibitors were submitted for evaluation by ABC Company. The inhibitors were evaluated in the stirred autoclave apparatus according to the protocol received from ABC Company. The products tested are listed in Section 2.1.

2. Test Conditions

2.1. Corrosion Inhibitors

<table>
<thead>
<tr>
<th>Product Name</th>
<th>Type</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>Oil Based</td>
</tr>
<tr>
<td>B</td>
<td>Water Based</td>
</tr>
<tr>
<td>C</td>
<td>Water Based</td>
</tr>
<tr>
<td>D</td>
<td>Water Based</td>
</tr>
</tbody>
</table>

Table 1 – Batch Corrosion Inhibitors

2.2. Brine

Synthetic brine was used for testing. It was prepared based on the water analysis supplied by ABC Company. The composition of the synthetic brine used is shown below.

<table>
<thead>
<tr>
<th>Sodium (mg/L)</th>
<th>Potassium (mg/L)</th>
<th>Calcium (mg/L)</th>
<th>Magnesium (mg/L)</th>
<th>Barium (mg/L)</th>
<th>Chloride (mg/L)</th>
<th>Bicarbonate (mg/L)</th>
<th>Sulphate (mg/L)</th>
</tr>
</thead>
<tbody>
<tr>
<td>2236</td>
<td>31</td>
<td>17</td>
<td>6</td>
<td>4</td>
<td>1021</td>
<td>3882</td>
<td>1</td>
</tr>
</tbody>
</table>

Table 2 – Water Analysis

The synthetic brine was prepared fresh prior to testing. The brine was pre-purged with CO$_2$ for a minimum of 2 hours and the pH was measured to be 6.21.

2.3. Autoclave Test Apparatus

The autoclaves used by Cormetrics Limited are constructed of Hastelloy 276-C and have a capacity of approximately 300mL. The tests were carried out with 250 mL of synthetic brine in each cell (approximately two-thirds full).
A three-electrode assembly is suspended from the lid of the autoclave, keeping the bottom clear for a Teflon-coated magnetic stir-bar. The configuration of the electrodes is a closely spaced equilateral triangle, with each cylindrical electrode having a 0.25” x 1.5” geometry. The reference electrodes are made from Hastelloy 276-C, while the working and counter electrodes are 1018 carbon steel. The electrodes are solvent rinsed and weighed prior to the commencement of the test period. A surface area of 7.92 cm² has been used throughout for corrosion rate calculations.

The temperature of the fluid in the autoclave is sensed by a thermistor probe, held at the center of the cell by a Hastelloy sleeve. Charging of the autoclave is by means of an offset Hastelloy tube, fitted with a pressure gauge and sour-service needle valve. Each cell is also equipped with a pressure relief valve which is used when purging the test liquids directly in the cells.

LPR measurements were obtained at 30-minute intervals by connecting each cell to a Gamry PC4-300 potentiostat and controller, via a Gamry ECM8 multiplexer. Data acquisition was by means of Gamry’s DC105 software package.

2.4. Batch Corrosion Inhibitor Application

A (the oil based product) was tested at 20%, by volume, in kerosene. The electrodes were immersed in the solution for 10 s followed by a minute of drying time and then two 1 minute rinses in purged brine.

The water based products were applied by immersing the electrodes in a 20%, by volume, solution of inhibitor in purged brine for 10 seconds. The electrodes were then rinsed in the same fashion as above: 1 minute drying time and two brine rinses of one minute each.

The electrodes for the blank cell were also rinsed in the same manner as described above.

2.5. Autoclave Pressuring

Once the cells had been sealed they were purged with carbon dioxide for an additional 15 minutes to remove any oxygen that may have been introduced in the filling process.

The autoclave cells were then filled with 173 psi of a gas mixture that was 1.5% H₂S in nitrogen and with 64 psi of CO₂. These pressures were used to match the target levels of 7.37% CO₂ and 3000 ppm of H₂S.
Finally each autoclave was filled to the total pressure of 800 psi with nitrogen.

Once pressurized, the autoclave cells were then placed inside individual heating mantles and brought to 32°C via proportional temperature controllers. The fluids were stirred at a rate of 200 rpm for the entire test period. The Gamry instrument was then programmed to start collection of LPR data.
3. Results Summary

3.1. Linear Polarization Resistance Data

![Graph showing LPR Data]

**Figure 1 - LPR Data**
Figure 2 - LPR Data; Expanded Scale
### 3.2. Gravimetric and Visual Analysis

<table>
<thead>
<tr>
<th>Inhibitor</th>
<th>Weight Loss (mg)</th>
<th>Weight Loss Corrosion Rate (mpy)</th>
<th>Visual Description</th>
<th>Pit Depth (mils)</th>
<th>Pit Rate (mpy)</th>
<th>Electrode Photo (Full)</th>
<th>Electrode Photo (Close-up)</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>0.25</td>
<td>0.15</td>
<td>Shiny surface</td>
<td>N/A</td>
<td>N/A</td>
<td><img src="image1.png" alt="Electrode Photo" /></td>
<td><img src="image2.png" alt="Electrode Photo" /></td>
</tr>
<tr>
<td>B</td>
<td>12.10</td>
<td>7.46</td>
<td>Large etched patch over half of electrode surface</td>
<td>N/A</td>
<td>N/A</td>
<td><img src="image3.png" alt="Electrode Photo" /></td>
<td><img src="image4.png" alt="Electrode Photo" /></td>
</tr>
<tr>
<td>C</td>
<td>0.35</td>
<td>0.22</td>
<td>Shiny surface, few small etched spots, several fine pit initiation sites noted</td>
<td>N/A</td>
<td>N/A</td>
<td><img src="image5.png" alt="Electrode Photo" /></td>
<td><img src="image6.png" alt="Electrode Photo" /></td>
</tr>
<tr>
<td>D</td>
<td>1.05</td>
<td>0.65</td>
<td>Shiny, edge attack, few etched spots</td>
<td>N/A</td>
<td>N/A</td>
<td><img src="image7.png" alt="Electrode Photo" /></td>
<td><img src="image8.png" alt="Electrode Photo" /></td>
</tr>
<tr>
<td>Blank - Batch</td>
<td>18.25</td>
<td>11.24</td>
<td>General corrosion</td>
<td>N/A</td>
<td>N/A</td>
<td><img src="image9.png" alt="Electrode Photo" /></td>
<td><img src="image10.png" alt="Electrode Photo" /></td>
</tr>
</tbody>
</table>

**Figure 3 – Electrode Photographs**
3.4 pH Data

<table>
<thead>
<tr>
<th>Cell</th>
<th>Initial pH of Brine</th>
<th>Final pH Of Brine</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>6.21</td>
<td>6.24</td>
</tr>
<tr>
<td>B</td>
<td>6.21</td>
<td>6.29</td>
</tr>
<tr>
<td>C</td>
<td>6.21</td>
<td>6.17</td>
</tr>
<tr>
<td>D</td>
<td>6.21</td>
<td>6.40</td>
</tr>
<tr>
<td>Blank</td>
<td>6.21</td>
<td>6.42</td>
</tr>
</tbody>
</table>

Table 4 – pH Values

4. Discussion

4.1. Continuous Corrosion Inhibitors

- The LPR corrosion rate for the inhibitor-free cell (blank) started at 40 mpy but quickly dropped to below 10 mpy. The rate then increased slightly and was quite stable from this point on ending at a level of 15 mpy.

  The weight loss corrosion rate was 11 mpy and the test electrodes exhibited general corrosion.

- The oil based inhibitor; A was able to maintain low LPR rates for the entire test. The rate began at around 0.1 mpy and trended upward ending with an LPR rate of 0.2 mpy. The trend was very smooth and stable which indicates a strong inhibitor film on the metal surface.

  The weight loss corrosion rate was the lowest of any of the products at 0.15 mpy. The electrode surface was shiny with no corrosion damage apparent when viewed microscopically.

- The B product began with an LPR corrosion rate of 2 mpy but the protective batch film quickly broke down causing the corrosion rate to climb to a level higher than that of the blank for a portion of the test. The final LPR corrosion rate for this product was 6 mpy.
The weight loss corrosion rate was 7.5 mpy and the electrodes exhibited a very large etched patch that covered almost half the surface. These observations were consistent with the breakdown in film that was noted on the LPR graph.

- The C product exhibited the lowest LPR rates of all of the inhibitors evaluated. It began at 0.7 mpy but quickly declined to a very low level and ended with a final rate of 0.002 mpy.

Despite the very low general LPR corrosion rate that was noted above, the weight loss corrosion rate was 0.2 mpy. This may have been due to the higher initial corrosion rate or due to the appearance of some fine localized damage noted on the electrode surface. The appearance of any localized damage automatically downgrades the ranking of the inhibitor.

- The D product began with an LPR corrosion rate of 0.6 mpy. The rate remained under 1 mpy for the entire test period however it did fluctuate. The highest level was 0.98 mpy at 40 hours. The rate settled out after this point reaching a final LPR corrosion rate of 0.7 mpy.

The weight loss corrosion rate was 0.65 mpy. The electrodes exhibited a shiny surface with some edge attack and several small etched spots.
5. Conclusions

Based on testing performed in our laboratory under these conditions the water based products can be ranked as follows:

\[ \text{D} > \text{B} > \text{C} \]

The C product was ranked as the poorest performer due to observed pit initiation sites. While a pit depth could not be measured there was depth to the defects which led to them being classified as pitting as opposed to etching.

The oil based product, A, performed very well. It was able to maintain very low LPR corrosion rates, weight loss corrosion rates and virtually no corrosion damage was detectable on the electrode surface. We would recommend this as an excellent product under these conditions.

Sincerely,

Cormetrics Limited

____________________________________________________
Lab Manager

____________________________________________________
President

*Please note, all inhibitor samples and electrodes are stored for 6 months prior to disposal.*